



Cost-effective Clean Electrochemical Preparation of Ferric Chloride and its Applications



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Abstract

In this investigation, a green, cost-effective clean electrochemical process for preparing a highly concentrated ferric chloride (FeCl_3) solution has been proposed. Many operating conditions, affected the used anodic dissolution process for FeCl_3 production, were studied. Generating ferrous hydroxide electrochemically then dissolving it by hydrochloric acid and oxidized with chlorine free mixed oxidant, were proceeded steps to reach the production of our target ferric chloride. It is a green, clean, and novel process because it is environmentally and industrial safer than the traditional production technology. The most important studied experimental variables were applied like applied current density, sodium chloride concentration, electrolyte pH, at the most controlling operating parameter which is optimum electrolysis time. Experimental results revealed that optimum current density was 300 mA/cm^2 ; using 30 gm/L saline solution, electrodes gap distance 5 cm , and stirring rate 150 rpm at $\text{pH } 6.5$ for 270 minutes, respectively. At these optimal operating parameters, 40% FeCl_3 concentration can be prepared to consume 202 watts/liter of high quality with low capital, & operating costs. The economic indicators show that the running cost of production of 40% ferric chloride solution using the proposed cleaner electrochemical production technology will be $241.66 \text{ \$/ton}$ and the suggested price is $450 \text{ \$/ton}$ with a net profit of $156 \text{ \$}$ with high cash flow using the proposed green technology. The proposed methodology for FeCl_3 production reviews the importance of using cleaner production and sustainable development indicators.

Keywords: Ferric Chloride, Electrochemical generation, Cleaner production, Applications.

1. Introduction

At the time being, many investors are investing in the production of ferric chloride (FeCl_3) due to the low cost of raw materials. The importance of FeCl_3 production attributed to the increasing awareness of its use in the treatment of drinking water as a coagulant especially in developing regions which expect to raise its requirements during the forecast period that leads to promising growth in the market. Furthermore, ferric chloride attributed relatively lower cost with better performance characteristics than other water treatment coagulants like poly-aluminium chloride, ferrous sulfate, and alum which help in driving the obligation of FeCl_3 consumption and marketing as reported by Nicos, 2001 [1].

Aqueous solutions of FeCl_3 attributed numerous applications, such as flocculating agents for seawater pre-treatment prior desalination [2], feed additives

[3], wastewater treatment [4], soil treatment [5], drinking water flocculation [6], phosphate removal [7], heavy metals removal [8], printed circuits etching in electronic industry [9], ideal Lewis polymerization in organic synthesis, reduction, C-C bond formation, oxidative coupling, and others [10].

On the other hand anhydrous FeCl_3 or its hexahydrated form reacted as oxidant for bacterio-pyrophosphoride to produce ring-D oxidized chlorine in $>95\%$ yield [11]. FeCl_3 solution is considered one of the most important applications in both potable and wastewater treatment processes. The Mechanism of Ferric Chloride Preparation could be explained as follows:

Ferric chloride solutions can be produced by oxidation of ferrous chloride using active oxygen (O^{++}) or chlorine (Cl_2) or either by the dissolution of ferric oxide with hydrochloric acid. Preparation of iron solution is typically supplied with iron concentration with 10 to 14 weight percent because at

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iron concentrations greater than 15% by weight, precipitation may be result, especially when ferric chloride solution was subjected to relatively low temperatures at 0° C or less. Anhydrous ferric chloride may be prepared by a union of ferrous elements as stated in Wikipedia, 2020 [12] as follows:

- Dissolution of iron ore in hydrochloric acid to get FeCl₂
- Oxidation of FeCl₂ with chlorine
- Use of oxygen for oxidation of FeCl₂

Also, FeCl₃ can be produced by reacting hydrochloric acid with iron, but in small quantity, while FeCl₂ was transferred to FeCl₃ by oxidation using hydrogen peroxide [13]. Recently; companies are supplying FeCl₃ from iron with chlorine gas or from iron oxides, hydrochloric acid, and chlorine gas according to the following reactions: ferrous chloride is obtained by reaction of ferric chloride with iron according to the following reaction.

Also pickling solutions can also be recovered; these solutions consist of dilute ferrous chloride and hydrochloric acid. It is not possible to concentrate ferric chloride solutions since they decompose when heated. It is therefore necessary to improve concentrated solutions of chloride. For example, to obtain a 40% by weight solution of ferric chloride, it is necessary to start with a 34.25% solution of ferrous chloride if only chlorine is added.

It is necessary to first destroy the hydrochloric acid with powdered iron, to concentrate it and then to improve it, using chlorine gas as ferric chloride. This process is described in US Patent 4,066,748. US Patent 2,096,855 [14] describes a preparation of ferric chloride from iron and chlorine without using hydrochloric acid. Ferrous chloride reacts with chlorine gas to give ferric chloride, part is recycled to attack iron according to equation (1), and the corrosive properties of ferric chloride accelerate the dissolution of iron to ferrous chloride [15]



The ferrous chloride solution is filtered and oxidized to ferric chloride with chlorine gas as follows:



Iron oxides ore is a mixture of ferric and ferrous oxides. A treatment with 32% hydrochloric acid, followed by chlorination with chlorine gas, results in the formation of ferric chloride according to the following reactions:



On using chlorine gas, the industrial production process of ferric chloride is very corrosive which required special materials of the manufacturing equipment's with the addition of safety and environmental precautions. But looking for integrated preventative environmental for improved resource efficiency, minimization of risks, environmental impact, reduced waste, in organization's operations, lowering corrosively oxidizing agent of ferrous chloride and finally decreasing both capital and running cost of FeCl₃ production plants, it was found that one of the most promising desired oxidizing agents is the electrolytic generated mixed oxidant; (especially chlorine-free mixed oxidant).

The major component of the chlorine-free mixed oxidants is hydrogen peroxide and dissolved ozone. Hydrogen peroxide could be generated via electrolysis of sodium sulfate in an acidic medium as described by Chumakov et al, 2016 [16]. In addition, ferrous ions could be oxidized to ferric ion using hydrogen peroxide as represented by Davila et.al [18].

To fulfil a cleaner and sustainable production process should be preceded with the following parameters: changes in technology, varying operating practices, modifying in product design, reuse of wastes, and consequently changes in maintenance [17]. Based on the above literature review all the known production technologies are corrosive one and environmentally unsafe. We need novel cost-effective, easily controlled, environmentally friendly and with low capital and running cost production technology which achieved using the proposed green and cleaner electrochemical technology.

The main aim of this work is to develop a cost-effective green process to produce high concentrated ferric chloride solution through a cleaner production process including electrolytic generation of ferrous hydroxide which will be oxidized with hydrochloric acid and converted into ferric chloride through the addition of the electro-generated free chlorine mixed oxidant solution.

1. Experimental work

Electrochemical generation of FeCl_3 needs two electro cells as shown in Figures (1a & 1b)

a. Experimental set-up

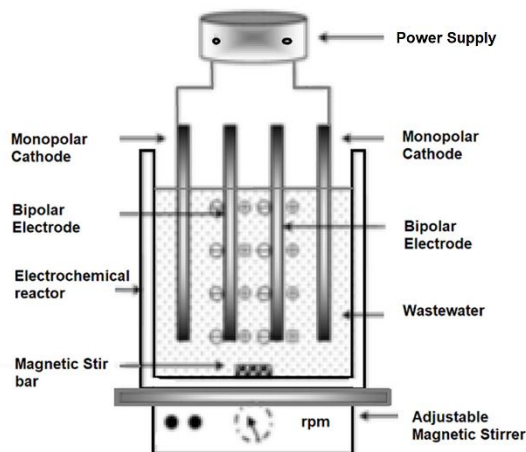


Figure (1a) Anodic dissolution cell

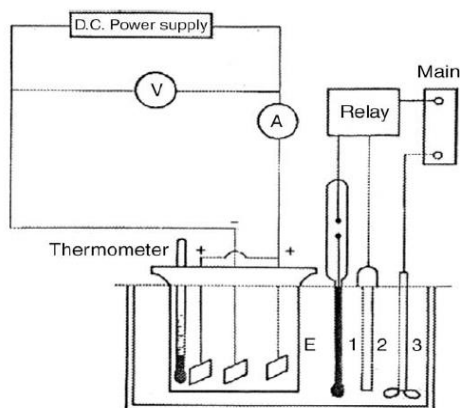


Figure (1b) Cl free mixed oxidant generation cell

Figure (1a) represents the iron anodic dissolution cell while Figure (1b) shows the chlorine generation cell for chlorine-free mixed oxidant solution to convert ferrous chloride to ferric chloride [20]. The iron dissolution cell consists mainly of iron anodes and cathodes maintained with D.C. power supply (model GP4303D LG precision Co. Ltd, Korea). Iron electrodes of (2 x 10) cm were mechanically polished with different grade emery papers, washed with distilled water, then rinsed in acetone, and finally dried in air stream.

2.2 Experimental procedure

The anode was weighed and inserted vertically in a midway position in a midway between the cathodes. The gap distance between each cathode and anode was 3 cm. The anodic dissolution cell was 1.25 liter volume and

filled with 1 liter saline solution for each experiment. All experiments were carried out under stirring while voltage and current were measured using a digital multi-meter; kyoritus model 1008, Japan. The dissolution experiments were carried out at ambient temperature 25°C. On starting the run; the pH and conductivity were adjusted to the desired values.

The conductivity of the solution was increased by adding the desired amount of the supporting electrolyte and dissolved completely, then the pH was adjusted to the desired value. Stirring was started up and direct current was applied for the required time then the system was switched off. Ferrous hydroxide separated by decantation and filtration. The dissolved iron was measured by the difference in anode weights before and after the dissolution process. The anodic dissolution efficiency was calculated from equation (6) as reported by Didukh et al, 2017 [19].

$$\text{Anodic diss. Eff.} = \text{wt. diss.} \times 100 / I (\text{amp.}) \times t (\text{sec}) \times \text{ECE} \quad (6)$$

Where: (ECE) is electrochemical equivalent of iron. The generated ferrous hydroxide was reacted with the stoichiometric amount of hydrochloric acid to be converted to acidic ferrous chloride solution. The solution was oxidized to 40% ferric chloride solution by the free chlorine mixed oxidant solution. Total iron concentration, ferrous and ferric ions, was measured according to Didukh et al, 2017 [19]. Figure (2) represents the block diagram for electrolytic preparation of ferric chloride solution

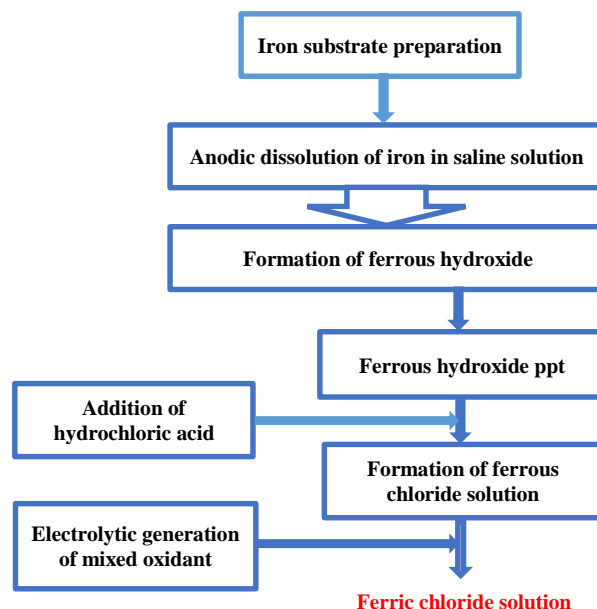


Figure (2) Block diagram for electrolytic preparation of ferric chloride solution

According to Shaarawy, et al, 2012, the chlorine-free mixed oxidant has been produced using electrolysis of sodium sulfate solution via the cell described in Figure (1b) and according to the procedure described by Shaarawy, et al. Rh/RhO/Ti DSA is the modified anode was used in the electrolysis process. The generated hydrogen peroxide during electrolysis was determined according to the stated analytical method [20]

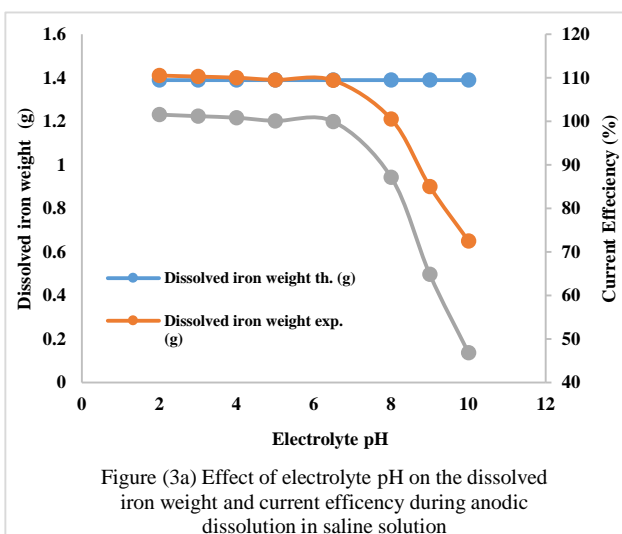
3. Results

Several operating parameters controlling the success of ferric chloride production via anodic dissolution process, which are mainly: electrolyte pH, sodium chloride concentration, applied current density, and electrolysis time (ET). ET is the most important parameter in the electrolytic generation of chlorine-free mixed oxidant to guarantee the generation of enough amount of hydrogen peroxide for complete conversion of ferrous chloride to ferric chloride.

3.1. Effect of pH in iron anodic dissolution

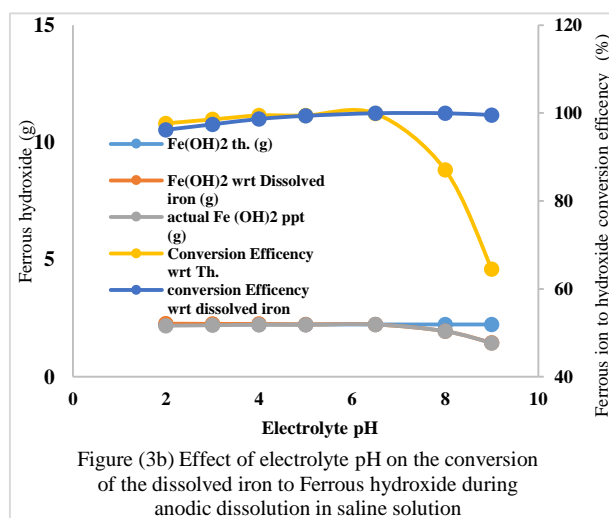
Figure (3a) shows the effect of the electrolyte pH on the generated dissolved iron weight, and the anodic dissolution current efficiency, while Figure (3b) represents the effect of using different pH on the conversion of generated dissolved iron ion to ferrous hydroxide precipitate. The operating conditions of the anodic dissolution process were sodium chloride concentration 10g/l, applied current density 100mA/cm², electrolysis time 60minutes, electrodes gap distance of 5cm, and stirring rate 150rpm.

The results from Figure (3a) revealed that: in the acidic range the dissolved iron ions generated from the iron anode exceed the theoretical value calculated according to Faraday's law, this could be attributed to



that: lowering pH values, two types of iron dissolution occur which are electrochemical dissolution in addition to chemical dissolution as a result of the presence of hydrochloric acid used for pH adjustment. At neutral pH, the generated dissolved ions may be typically equal to the calculated value. Increasing pH to the alkaline region, the amount of generated dissolved iron experimentally started to decrease due to electrode passivation.

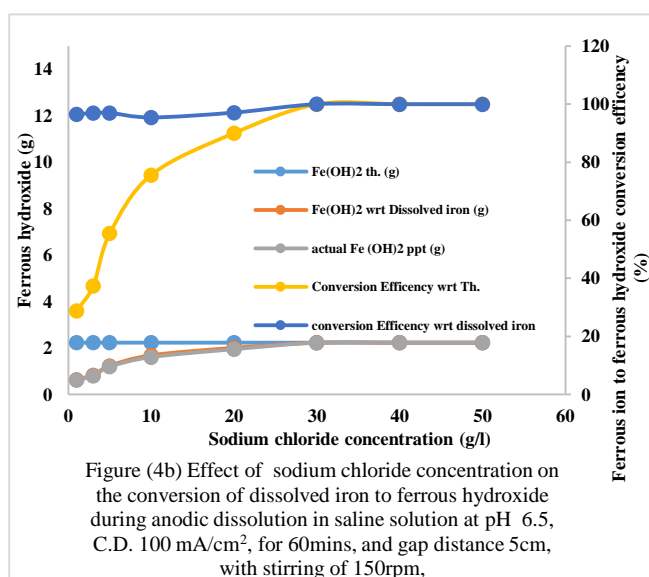
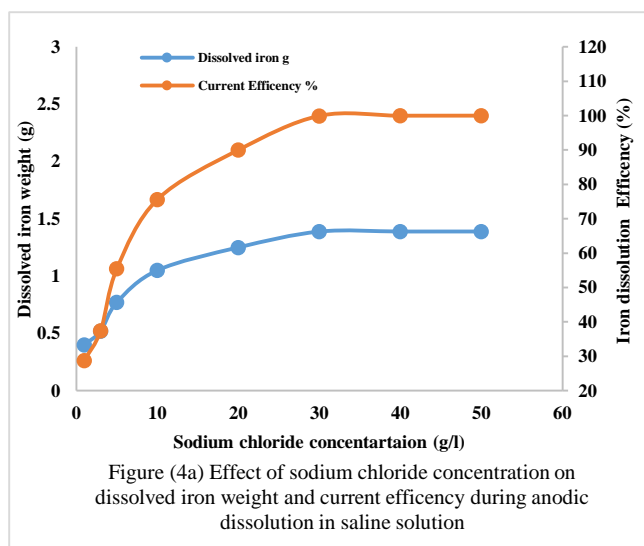
The results in Figure (3b) show that at low pH values the conversion efficiency from ferrous ion to ferrous hydroxide precipitate is lowered due to the presence of hydrochloric acid which reacts with ferrous ion to form ferrous chloride (green solution). As the pH value moved to the alkaline region, the conversion efficiency to precipitated ferrous hydroxide increased up to pH 8; the formation efficiency decreased due to the decrease of anodic dissolution efficiency. Based on the above results, a pH value of 6.5 was taken as optimum pH during the iron anodic dissolution process.



3.2. Effect of sodium chloride concentration

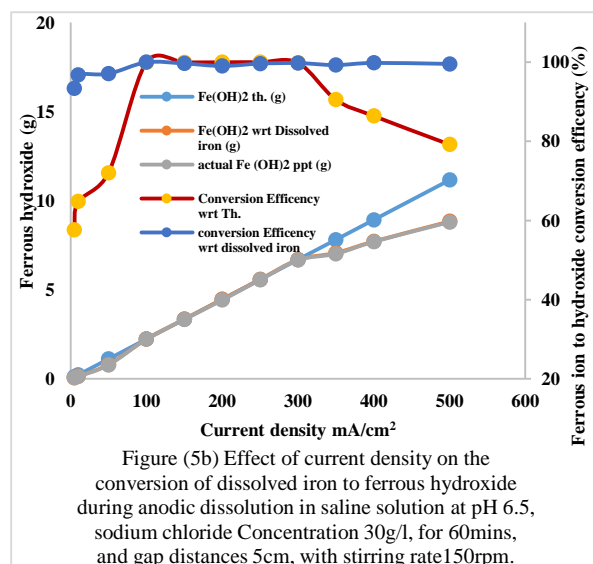
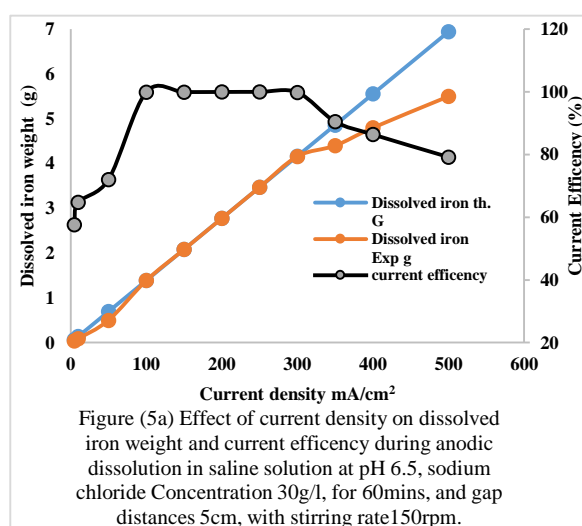
Figures (4a & 4b) show the effect of sodium chloride concentration on generated dissolved iron weight, anodic dissolution current efficiency, and the conversion of generated dissolved iron ion to ferrous hydroxide precipitate, respectively during the iron anodic dissolution process. At the following operating conditions: pH 6.5, applied current density of 100 mA/cm², electrolysis time 60mins, and electrodes gap distance 5cm, with stirring of 150rpm, results indicated that, with increasing sodium chloride concentration, the generated dissolved iron weight, the anodic dissolution current efficiency, and the conversion of the generated dissolved iron ion to

ferrous hydroxide precipitate increase reaching their maximum values at sodium chloride concentration 30g/l then no significance change obtained at higher concentration of sodium chloride. So 30g/l sodium chloride was taken as optimum electrolyte concentration during iron anodic dissolution process.



3.3 Effect of applied current density

The effect of applied current density on generated dissolved iron weight, anodic dissolution current efficiency, and conversion of generated dissolved iron ion to ferrous hydroxide precipitate, are respectively graphically represented in Figures (5a & 5b) during the iron anodic dissolution process.



These were experimentally carried out at pH 6.5, sodium chloride Concentration 30g/l, electrolysis time 60mins, and electrodes gap distances 5cm, with stirring rate 150rpm.

The results indicate that as current density increases the dissolved iron ions generated increases, while the current efficiency increases from current density 5mA/cm² till 100mA/cm² which is the maximum current efficiency, more increase of the applied current density the current efficiency is still constant till 300mA/cm² and after that current efficiency starts to decrease due to the formation of black oxide layer covering the iron anode. Figure (5b) shows that the conversion efficiency of ferrous ions to ferrous hydroxide reached its maximum at 300mA/cm² while increasing applied current density decreased the conversion efficiency. Based on the above results

applied current density of 300mA/cm² was taken as optimum.

3.4 Effect of electrolysis time

Figures (6a & 6b) represent the effect of electrolysis time on both iron anodic dissolution process and iron ion conversion to ferrous hydroxide, respectively at applied current density 300mA/cm², pH 6.5, and sodium chloride concentration 30g/l, electrodes gap distance of 5cm and stirring rate 150rpm. The results show that as the electrolysis time increases the generated dissolved iron ions and the formed ferrous hydroxide increase, while both the anodic dissolution current efficiency and conversion efficiency reached about 99% after 4 hours

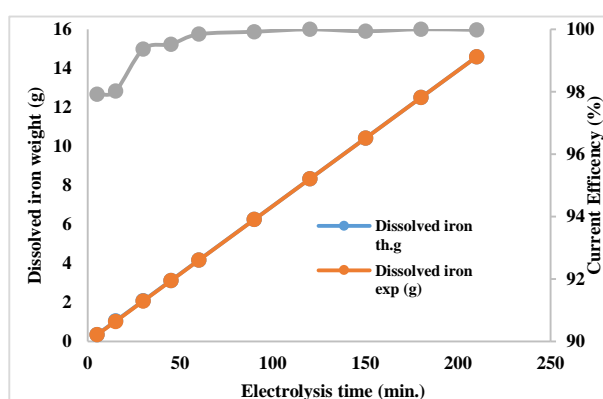


Figure (6a) Effect of electrolysis time on dissolved iron weight and current efficiency during anodic dissolution in saline solution

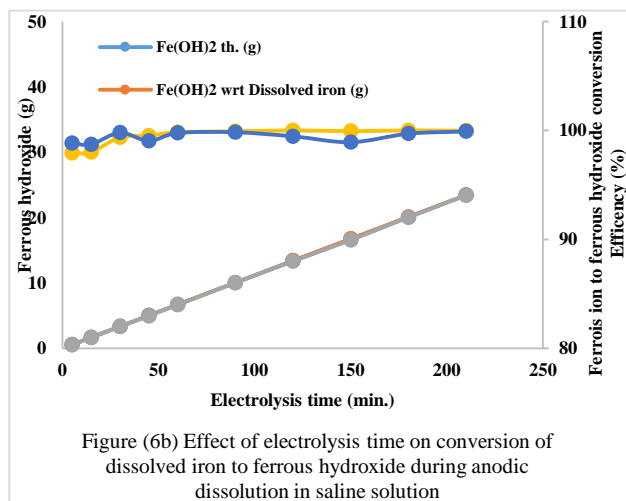


Figure (6b) Effect of electrolysis time on conversion of dissolved iron to ferrous hydroxide during anodic dissolution in saline solution

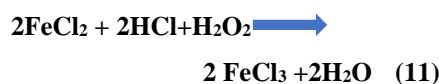
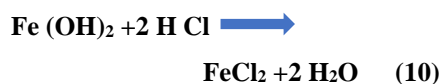
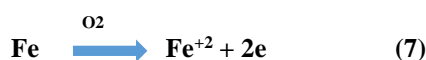
3.5 Effect of gap distance

With respect to the gap distance, it is found that the gap distance only affects the anodic dissolution voltage, at a gap distance of 2 cm the voltage is 2V while the dissolution efficiency is 85% due to the formation of a black layer of iron oxide. At 5cm the voltage is 3V but the dissolution efficiency reached

the maximum, while at a gap distance of 7 cm the dissolution efficiency decreases so the optimum gap distance of 5cm is 5V to reach optimum current density 300mA/cm². The process voltage only affects consumed electric power which will increase the cost, so 5cm was taken as the optimum gap distance.

From experimental results, the optimal conditions for iron anodic dissolution in saline solution are pH 6.5, applied current density 300mA/cm², sodium chloride concentration 30g/l, gap distance 5cm, stirring rate 150rpm, and electrolysis time as required. To prepare one liter of 40% ferric chloride we need to generate 112gm of dissolved iron using the anodic dissolution process. So at the above optimum conditions of dissolution using three iron anodes of area 30cm² were used to generate the required amount in about 4hrs.

After the preparation of the electro-generated ferrous hydroxide precipitate, it is washed with running tap water, then distilled water and reacted with the stoichiometric amount of hydrochloric acid (33%) to obtain the acidic ferrous chloride solution then by further oxidization using electro-generated chlorine-free mixed oxidant. The following equations show the mechanism of formation of ferrous chloride solution:



According to Shaarawy et al. [20], optimal conditions for the electrolytic generation of mixed oxidant were: pH 2.7, applied current density of 250mA/cm², electrolysis temperature of 25°C, electrolysis time 2hrs, cathode to anode area ratio of 1:1, gap distance between the anode and cathode of 3cm, rate of stirring of 300rpm, and NaCl concentration of 35g/l.

In this study, the sodium chloride electrolyte was replaced by sodium sulfate to get chlorine-free mixed oxidant with a major production of hydrogen peroxide. Titanium-based rhodium/ rhodium oxide (Rh/Rho) was thermally activated modified electrode with an area of 4cm*15cm*0.5cm (60cm²) was used as anode and high grade glassy graphite sheet was used as cathode.

After applying D.C current for a definite time the current switched off and the obtained mixed oxidant is added to the acidic ferrous chloride solution and left for 24 hrs. The produced solution is subjected to ferrous and ferric ion analysis to evaluate the conversion efficiency of ferrous chloride to ferric chloride. The test is carried out on a sample of 100ml of acidic ferrous chloride.

Figure (7) shows the effect of electrolysis time for the generation of chlorine-free mixed oxidant on the conversion efficiency of ferrous to ferric chloride, at the above-mentioned mixed oxidant operating conditions. The results revealed that complete conversion of ferrous chloride to ferric chloride in its solution is reached at electrolysis time of 270 minutes with mixed oxidant generation efficiency of 90% at 3 volts. The consumed electric power for complete conversion can be calculated according to the following equation:

$$\text{Power} = \text{Electrolysis Time (hrs)} \times \text{DC Current (A)} \times \text{Operation Voltage (V)}$$

From calculations, the required power for mixed oxidant generation is 202W to prepare 1 liter of 40% ferric chloride solution.

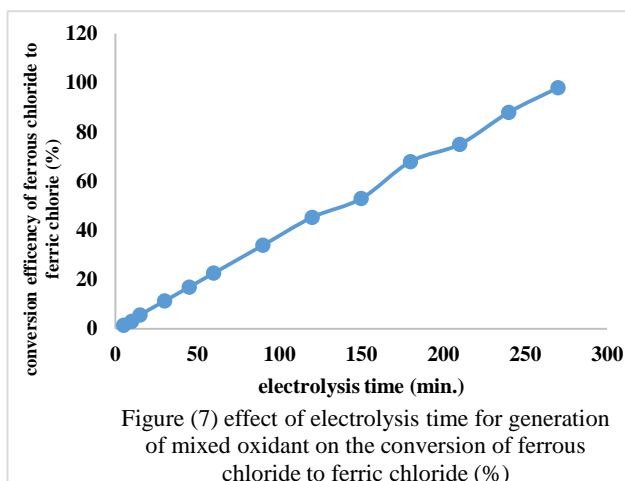


Figure (7) effect of electrolysis time for generation of mixed oxidant on the conversion of ferrous chloride to ferric chloride (%)

4.1 Unit description

This unit consists mainly of:

- **Sodium chloride electrolyte preparation tank** where the sodium chloride solution is prepared according to the required concentration
- **Iron anodic dissolution cell**
It is an anodic dissolution cylindrical cell of 500L volume constructed from PVC with iron anodes and St.St304 cathodes of active surface area 2500cm² and 250L/hr. flow rate.

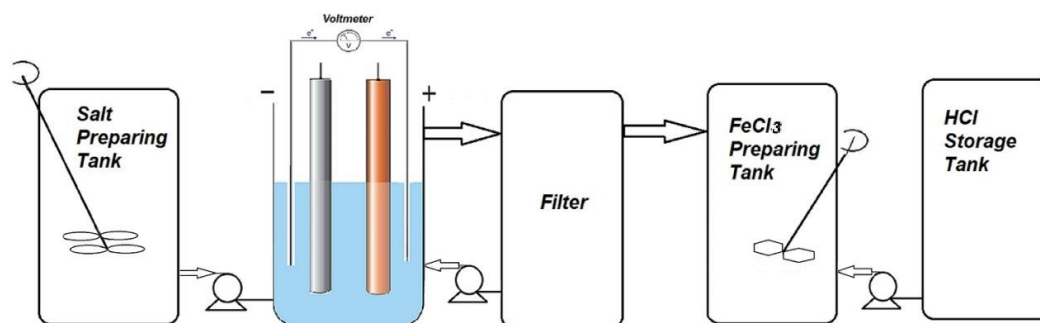


Figure (8) Flow diagram of the proposed pilot plant for FeCl₃ production

4. Pilot scale treatment unit

Based on bench scale results a production pilot plant unit of one ton per day capacity for the green generation of 40% ferric chloride solution is preliminarily designed to investigate the bases of the industrial-scale production unit. Figure (8) shows the flow diagram of the proposed pilot plant for the production of 40% ferric chloride solution according to the suggested technology.

- **Power supply**
It consists of a DC power supply; with a digital display and variable current control from 0-750A and variable voltage control of 0-100V.
- **Filtration unit**
To filtrate and collect the formed ferrous hydroxide cake, a PVC unit is made of PVC 50kg/hr. production capacity is needed.
- **Ferrous chloride preparation tank:**
It is a cylindrical PVC tank with a total capacity of 500 L provided with a mechanical stirrer to

react ferrous hydroxide with hydrochloric acid for acidic ferrous chloride solution generation.

- **The Chlorine-free mixed oxidant generation unit**

Mixed oxidant generation cell of cylindrical shape and 100L capacity with a flow rate of 250 L/hr, sodium sulfate was used as an electrolyte and Rh/RhO/titanium modified electrodes were used as anode and st.st304 was used as cathode. The cell is powered by a DC power supply of 0-100A current control and 0-24V voltage control.

- **Ferric chloride preparation tank:**

This is a PVC cylindrical shape cell of one-ton capacity in which the acidic ferrous chloride solution is mixed with the electro-catalytically generated chlorine-free mixed oxidant and stirred for 15minutes then left for 24 hours for reaction completion.

- **Feeding and transferring pumps,** control panel auxiliaries such as valves, and gages with the addition of piping system.

4.2 Operating conditions

Figure (9) shows the material balance of electrochemical production of 40% ferric chloride unit with the operating conditions according to the proposed technology.

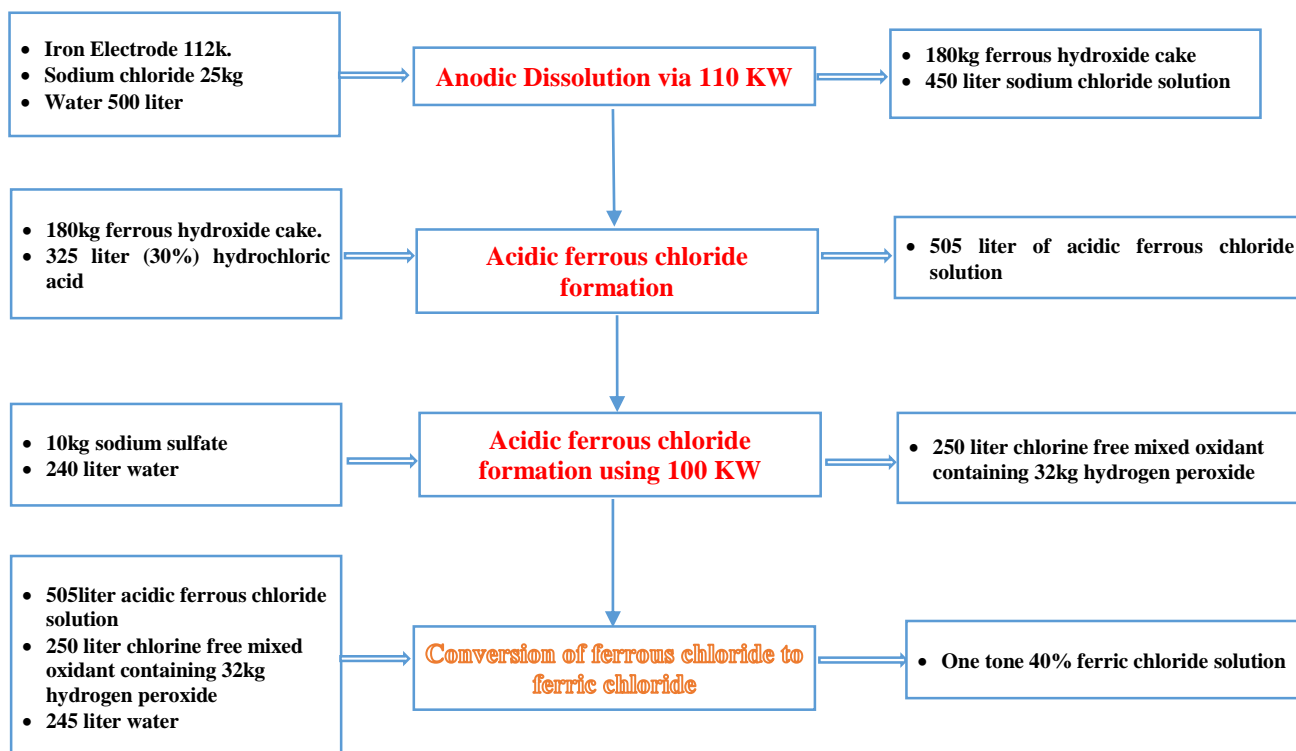


Figure (9) Material balance of electrochemical production

5. Discussion

Nowadays no business can succeed in an economic and social vacuum. Enterprises can achieve enduring success only by meeting the needs and the expectations of customers, investors, employees, and the greater society. Cleaner and sustainable development demands attaining the three-dimensional balance between economic growth and social and environmental responsibility [21].

5.1 Product quality

The obtained ferric chloride solution (40%) according to the proposed green electrochemical production technology was subjected to chemical analysis and it was found that the solution contains 11.2% of dissolved ferric iron with a brown clear solution without any precipitation and no other metals were found within the generated solution.

5.2 Power consumption

The proposed pilot system will consume electricity for the successive production of 40% ferric chloride solution treatment steps according to the following Table (1):

Table (1) The proposed system electricity

| Treatment Stage | Electricity Consumption, KW/ton |
|---------------------------------------|---------------------------------|
| Iron electrochemical dissolution cell | 110 |
| Mixed oxidant generation | 100 |
| Others | 5 |

As it is clear from the power consumption values the total electrical energy consumed during the production of the 40% ferric chloride solution is about 215 KW/ton. The electricity cost in Egypt is about 0.1\$ per KWhr.

5.3 Running cost

The running cost of the proposed system with respect to the cubic meter of 40% ferric chloride solution is illustrated in Table (2).

Table (2) shows that the running cost of production of 40% ferric chloride solution using the green electrochemical production technology will be 241.66\$ and the suggested price is 450\$ while the price of the 40% ferric chloride solution in china is 350\$ and when reaches Egypt it will cost about 450\$

Table (2) Running cost of the proposed system

| Treatment Stage | Unit | Q/ton | Unit price \$ | Cost \$ |
|--------------------------------------|----------------|-------|---------------|---------|
| Iron | kg | 112 | 0.8 | 89.9 |
| Sodium Sulfate | kg | 25 | 0.1 | 2.5 |
| Hydrochloric acid 33% | liter | 325 | 0.3 | 97.5 |
| Water including cleaning and washing | m ³ | 2 | 0.28 | 0.56 |
| Electricity | KW | 215 | 0.04 | 8.7 |
| Labour | | | | 15 |
| Transportation | | | | 15 |
| Maintenance | | | | 0.5 |
| Depreciation | | | | 7 |
| Others | | | | 5 |
| Total running cost | | | | 241.66 |

Table (3) Comparing proposed technology and traditional technology

| Item | Proposed System | Traditional system |
|--------------------------------|--------------------|-------------------------|
| Process production time (hrs.) | 8 | 72 |
| Power consumption K.W./ton | 215 | 50 |
| Production cost \$/ton | 241.66 | 160 |
| Product quality | High quality | Low quality |
| Sludge | No sludge obtained | High amount |
| Process corrosively | No corrosion | Very corrosive |
| Air toxicity | No toxicity | Very toxic chlorine gas |
| Safety | Very safe | Industrial unsafe |

Also the presence of sludge in the traditional production process whiles no sludge at all in the proposed green electrochemical production technology.

and with overhead and taxes of 45% will be sold in Egypt with a price of 750\$. The table shows that the global profit is about 208\$ and the tax is about 25% of the profit, which indicates that the net profit will be of 156\$ per ton. These results prove that the green electrochemical production of ferric chloride is a promising technology where this technology overcomes the bad environmental and safety impacts. And also have high cash value with promising economic indicators.

5.4 Comparison between proposed technology and traditional biological technology

Table (3) shows the comparison between the proposed clean and green electrochemical production of ferric chloride and the traditional chemical production technology. As it is clear that the proposed green electrochemical production technology is more environmentally and industrial safer than the traditional production technology where no air emission is obtained with regard to the very toxic and polluted air with a high concentration of chlorine gas obtained at the traditional production technology. Also, the obtained product is of high analytical grade quality while the traditional product is of low-quality grade.

6. Conclusions

A hybrid system of anodic iron dissolution, chemical acidification, and oxidation with chlorine free electro-generated mixed oxidant was used as advanced cost effective, green and cleaner electrochemical production technology of 40% ferric chloride solution to be used in water treatment coagulation process and printed circuits leaching in electronic industry. The proposed integrated system presents a successful process for obtaining high-quality ferric chloride solution with low capital and running costs. The proposed system was designed based on operating conditions of applied current density 300A/cm²; 30gm/L electrolyte concentration, gap distance 5cm, and stirring rate 150 rpm at pH 6.5 for 270 minutes. The economic indicators show that the running cost of production of 40% ferric chloride solution using the proposed cleaner electrochemical production technology will be 241.66\$ and the suggested price is 450\$ with a net profit of 156\$ which means high cash will be return using this technology. Also, a high cash flow product was produced from the proposed process. As a whole, the proposed system is one of the promising systems for the production of ferric chloride solutions.

Financial and Ethical disclosures:

No funding, no conflict of interest, and no used animals.

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